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Failure Analysis and Resin Evaluation of IUS SRM-1 92-inch Prototype Motor Case

Prepared by R. W. FILLERS, J. F. WARD, and P. K. GRANT
Materials Sciences Laboratory
The Aerospace Corporation
El Segundo, Calif. 90245

28 September 1979

Interim Report

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W. R. Shaffer, Colonel, USAF
Project Engineer

FOR THE COMMANDER

J. D. Mirth, Colonel, USAF
Deputy for Space Launch Systems

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The IUS SRM-1 92-in. diameter prototype Kevlar/epoxy motor case failed catastrophically at approximately 50 percent design load during hydrostatic burst testing on 18 October 1978. The Aerospace Materials Sciences Laboratory has carried out a failure analysis of the SRM-1 prototype motor case, briefly looked at two 18-in. subscale development vessels corresponding to SRM-1 and SRM-2, and carried out a preliminary evaluation of the epoxy resin system used for winding the motor case.

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ABSTRACT (Continued)

Examination of the three motor cases and preliminary examination of the resin system suggest that poor adhesion is obtained between the Kevlar fiber and this epoxy resin and further that this resin system gels in three to four days at room temperature. The result of poor adhesion is not known but is expected to be detrimental to case performance in the dome regions. However, excessively long winding times (on the order of six days), coupled with the short gel times of an extremely brittle resin system plus a lack of temperature control during winding, result in an extremely nonuniform Kevlar/epoxy composite. Since the local strains incurred through both premature gellation and thermal excursions during manufacturing cannot be relieved in a brittle matrix, premature failure can occur in a stepwise fashion at loadings well below design expectations, especially if the composite layers are not uniformly loaded.

PREFACE

The authors wish to acknowledge the contributions of C. N. Su for specimen preparation and optical microscopy, K. E. Wrightsel for the scanning electron microscopy, and R. A. Shenk for the differential scanning calorimetry. Technical requirements and general liaison with the IUS Program was provided by E. Y. Robinson.

CONTENTS

FIGURES

TABLES

I. INTRODUCTION

The IUS 92-in. SRM-1 prototype Kevlar/epoxy motor case failed at approximately 50 percent design load during final hydrostatic burst testing. This failure is thought to be related to the partial collapse of the plaster mandrel during winding of the case. This problem was "solved" by patching, or filling the low (flat) spots in the liner with a trowelable material prior to winding. All this results in nonuniform filament tension in the final case, which in turn prevents uniform loading of the Kevlar filaments. Since Kevlar fibers exhibit a linear stress-strain relation to failure, nonuniform loading is aggravated and leads to stepwise premature failure of the structure.

This investigation was directed principally toward the condition of the failed case as observed with both optical and scanning electron microscopes (SEM) and a brief preliminary study of the resin system. The purpose of the resin study was to test the hypotheses drawn from the microscopic observations.

II. FAILURE ANALYSIS

The failure analysis consisted of utilizing both optical and scanning electron microscopy to investigate the condition of the SRM-1 and SRM-2 subscale 18-in. vessels and the SRM-1 92-in. prototype motor case.

Figures 1 and 2 show typical cross sections of the SRM-1 and SRM-2 subscale 18-in. vessels, which were wound as part of the case development program. (The SRM-1 vessel was fabricated using wet winding procedures, and the SRM-2 vessel was fabricated using a wet "pre-preg" version of the same system.) The clean separation between fiber and resin evidenced in both figures indicates poor wetting, or adhesion, between the Kevlar fibers and the epoxy resin. Small pieces of poorly bonded resin are present in both figures, but particularly in Fig. 2.

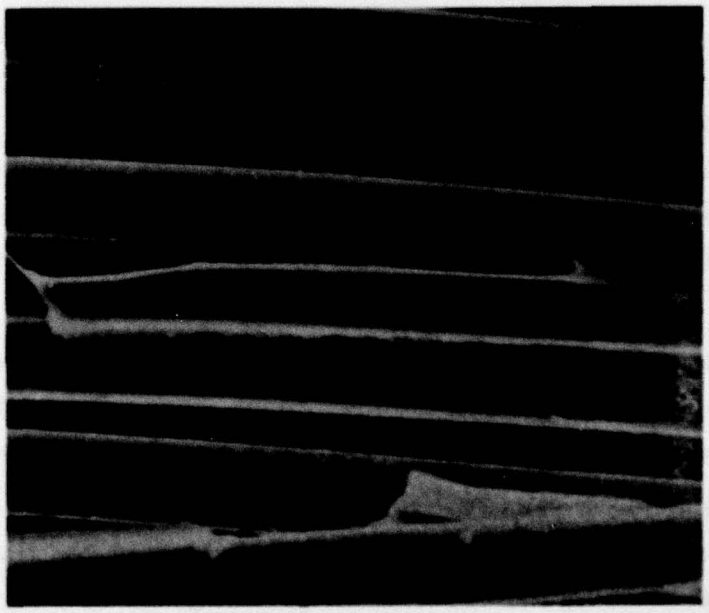
Figure 3 shows polished cross sections of the SRM-2 subscale vessel, taken at 50× (with the optical microscope). The two cracks running across the bottom of the pictures are delaminations, which occurred when the case burst. Note the poor distribution of fibers in the various wraps, or layers, and the resulting voids. The thickness of the inner wrap is approximately 0.010 in., whereas the outer wrap varies between 0.006 and 0.014 in. Also note that no fibers are split along the length of the crack.

Figure 4 is a schematic of the SRM-1 92-in. prototype motor case, showing the regions from which the specimens were taken. Both the SEM and optical microscopes were used to analyze these specimens.

Figure 5 shows a cross section of the case at Station 1. The region marked green rubber shows the material used to patch the liner (black rubber) prior to winding the case. This patching was used to fill the low spots and recover the contours of the discrepant plaster mandrel and rubber liner. Figure 5 also shows many radial cracks in the hoop wraps, which were observed prior to pressure testing. (These cracks were also noted

(a) 35 x

0.5 mm



(b) 1000 x

10 μm

Fig. 1. SRM-1 Subscale 18-in. Vessel

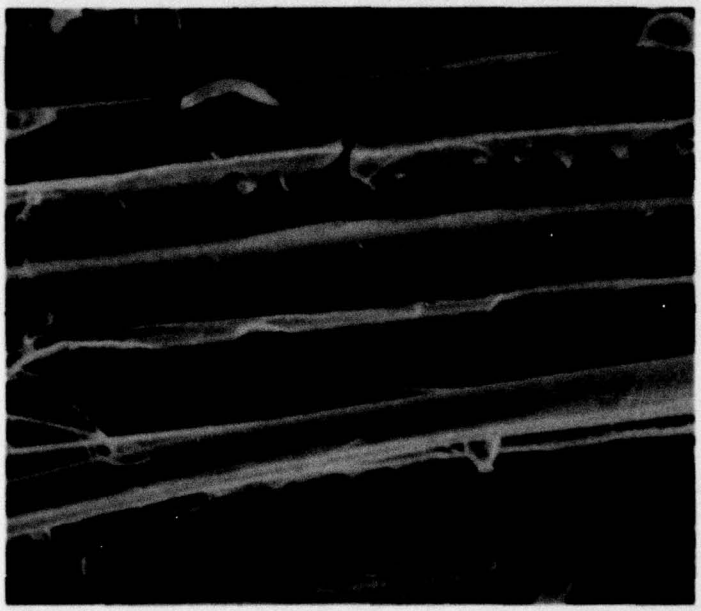
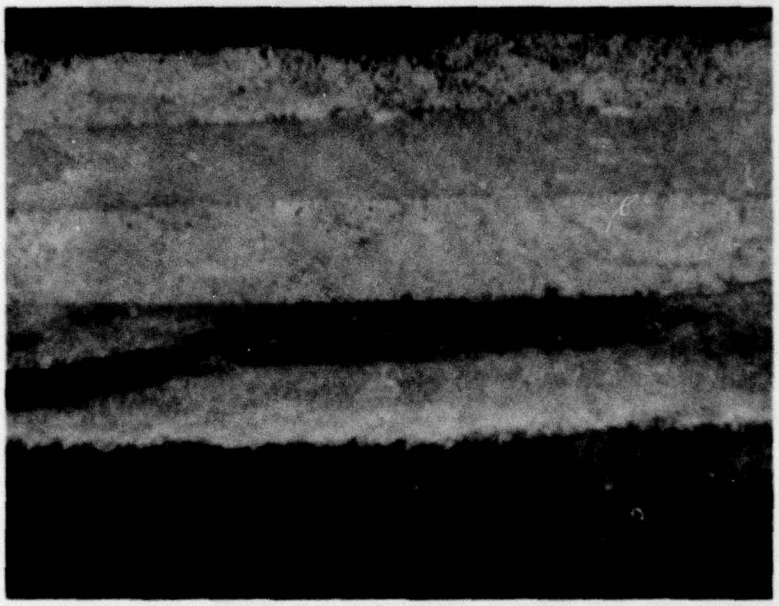


Fig. 2. SRM-2 Subscale 18-in. Vessel

a) 50 x: BRIGHT FIELD ILLUMINATION



b) 50 x: CROSS POLARIZED

Fig. 3. SRM-2 Subscale Vessel: Polished Cross Sections

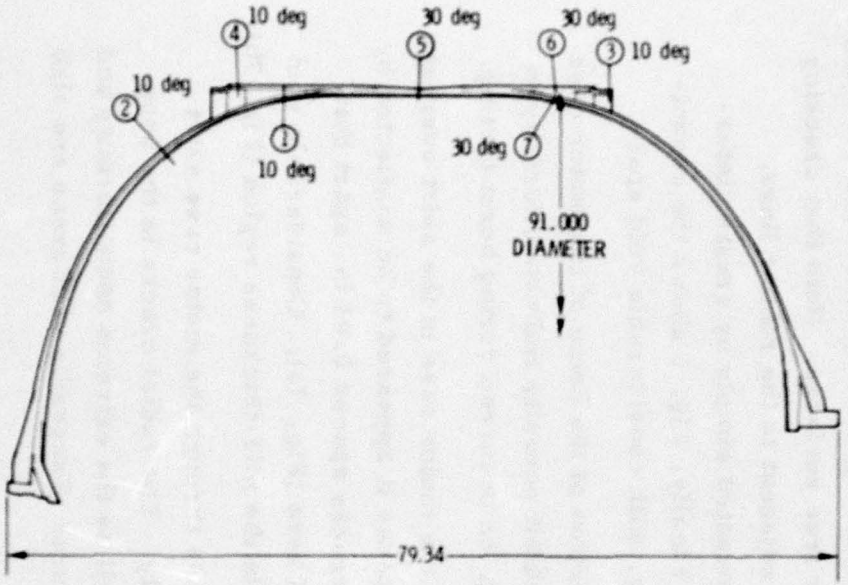


Fig. 4. SRM-1 92-in. Prototype

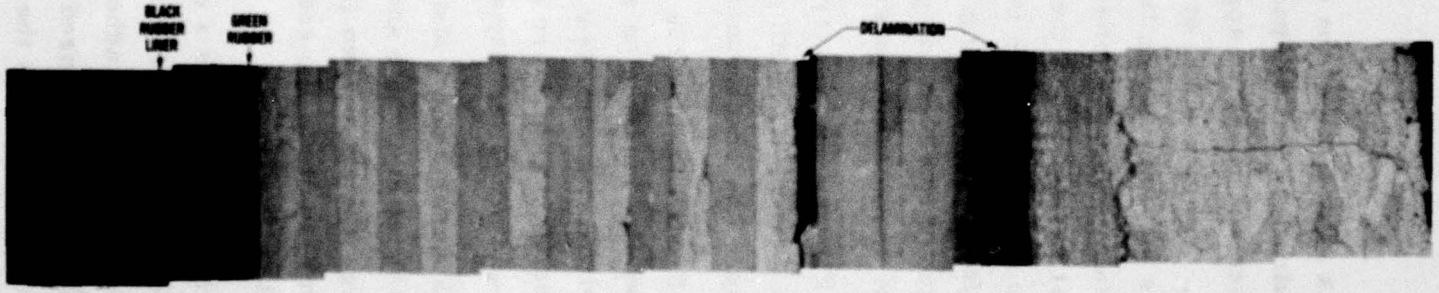


Fig. 5. Cross Section of Prototype at Station 1

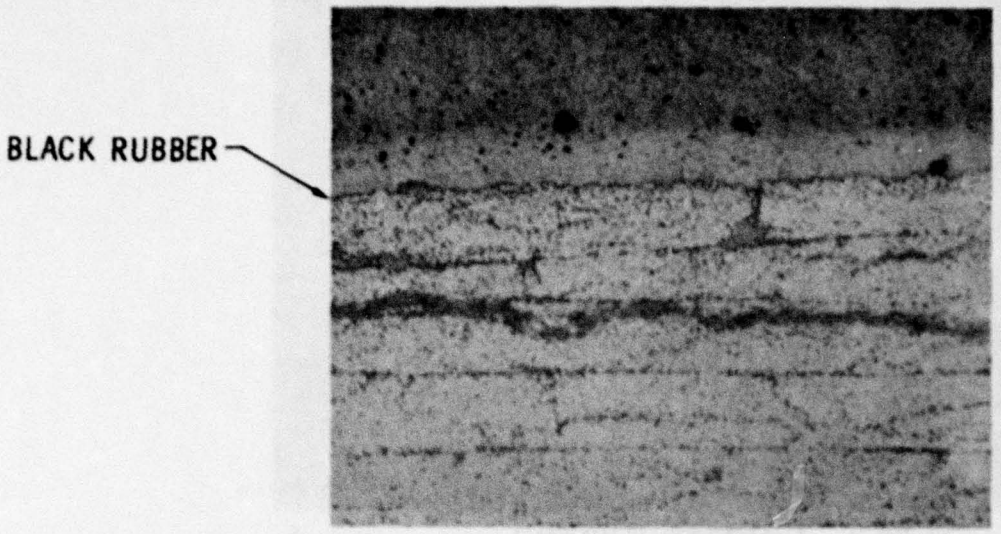
in the x rays taken of the case prior to hydrostatic testing.) They are approximately evenly spaced every 0.04 in. in all the outer hoop wraps observed and result in approximately one percent strain relief in the hoop wraps. The cracking, or "crazing," probably resulted from the thermal strains incurred during the cure cycle and indicates how highly strained the case was. Since the inner portions are held together by the outer, cracking is most pronounced at the boundaries or free surfaces. Note that cracking is also evidenced at the inner boundary adjacent to the rubber liner. Therefore, local crazing may not be prevented simply by greater interspersion of the hoop and polar layers. Finally, Fig. 5 shows the nonuniformity and nonhomogeneity in wrapping, with considerable void space.

Figure 6 shows a typical cross section of the dome of the motor case region (Station 2). Both photographs exhibit porosity and nonuniformity in winding. The crack running across Fig. 6a occurred during burst testing.

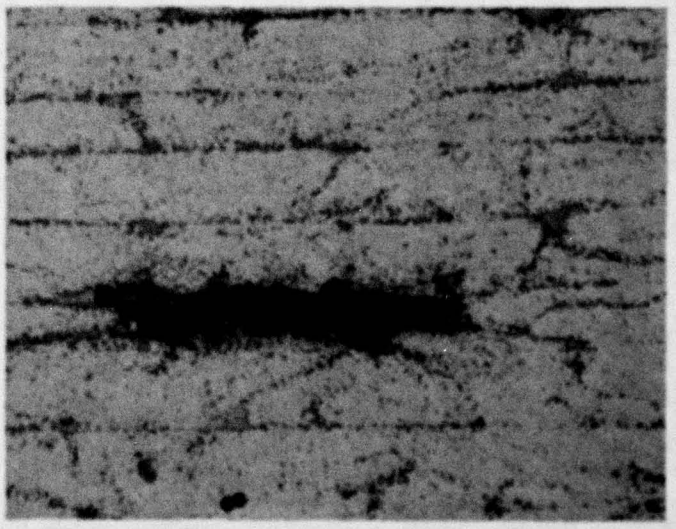
Figure 7 shows the appearance of the motor case in the skirt overhang (Station 3). This region was chosen because it appeared to be unaffected by the hydrostatic burst test. The radial cracks spaced 0.04 in. apart that were noted in Fig. 5 are also evidenced here (Fig. 7a). Considerable void space and nonuniformity are evidenced in the mid-thickness region (Fig. 7b).

Figures 8 and 9 show cross sections through the motor case skirt and dome at stations 6 and 7 respectively. The radial cracks in the hoop windings are again seen (Fig. 8), as well as the extreme nonuniformity and nonhomogeneity in the composite structure. Excessive void areas are also evident in Fig. 8.

Figure 10 shows a cross section of the Y joint taken at 30 deg aft. A high degree of nonuniformity is evidenced, especially in the layer adjacent to the rubber and the adhesive film laminate. Hoop wrapped filler is seen over the rubber plug used to correct large contour mismatch. Additionally, as noted previously in the other regions of the case, extensive voids were observed in both composite and adhesive.



a) 50 x: RUBBER LINER / CASE INTERFACE — 0.2 mm

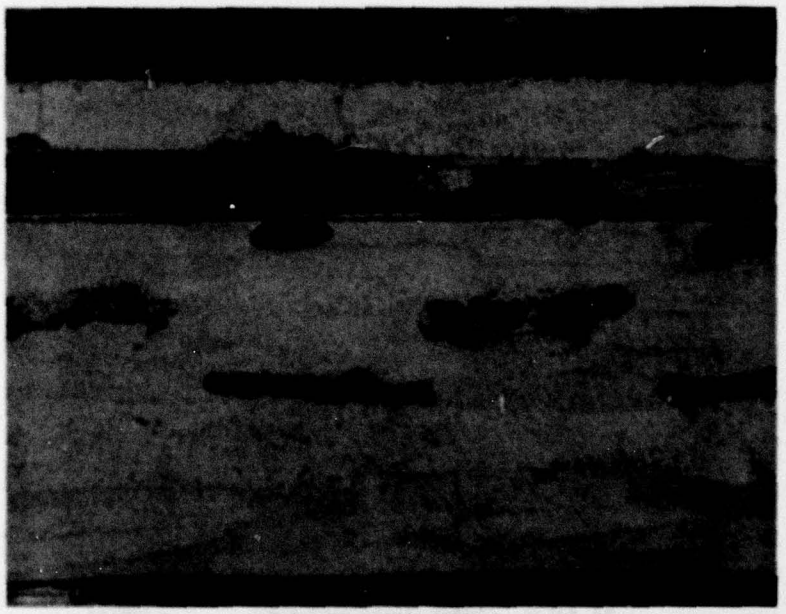


b) 50 x: MID-THICKNESS REGION — 0.2 mm

Fig. 6. Cross Section of Prototype at Station 2

a) 50 x: SKIRT/OUTBOARD REGION

0.2 mm



b) 50 x: MID-THICKNESS REGION

0.2 mm

Fig. 7. Cross Section of Prototype at Station 3

Fig. 8. Cross Section of Prototype at Station 6 (skirt only)

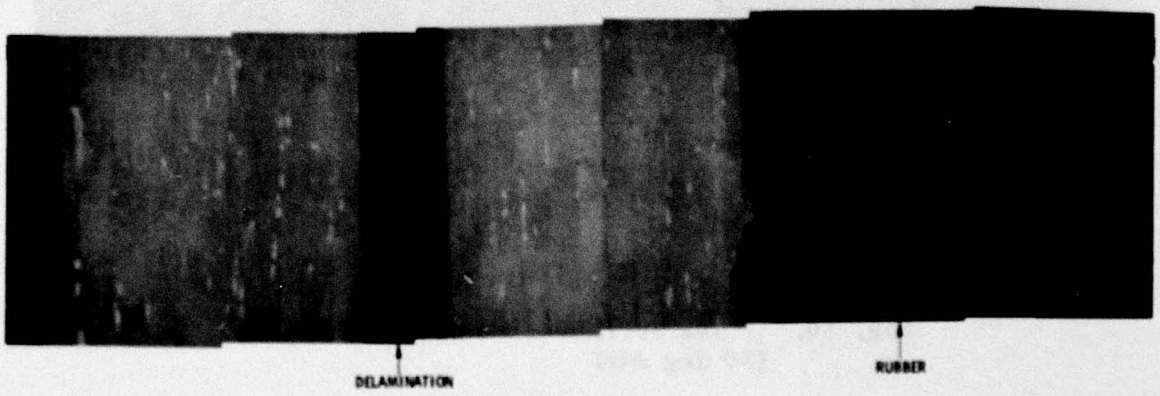


Fig. 9. Cross Section of Prototype at Station 7.

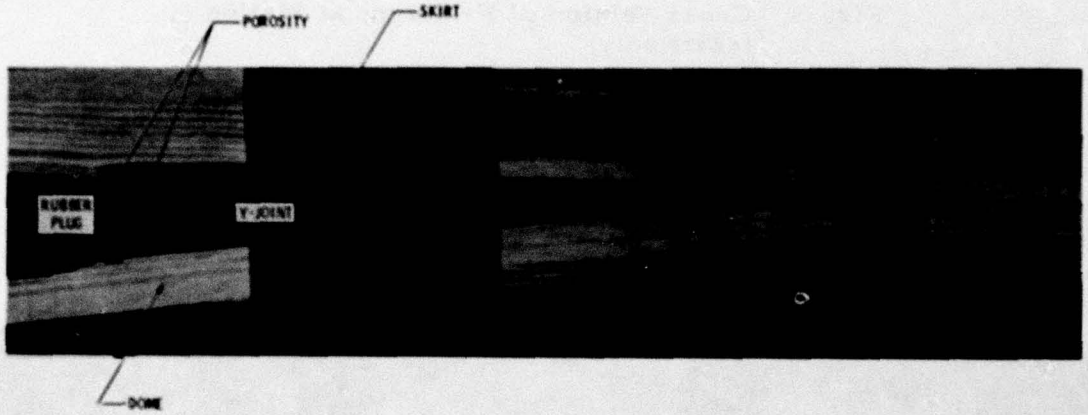


Fig. 10. Cross Section of Prototype Y Joint (30 deg Aft)

In addition to the optical microscopy of the polished specimens reported above, specimens were removed from the case dome at Stations 2, 3, and 4 and examined under the SEM. The only evidence of fiber wetting was observed in the skirt region in (Fig. 11a). This good adhesion was quite local and was not evidenced in any other region of that particular specimen nor in any other portion of the case. In general, all regions of the motor case observed with the SEM were identical to that shown in Fig. 11b.

The observations made during this portion of the investigation are that the case exhibits:

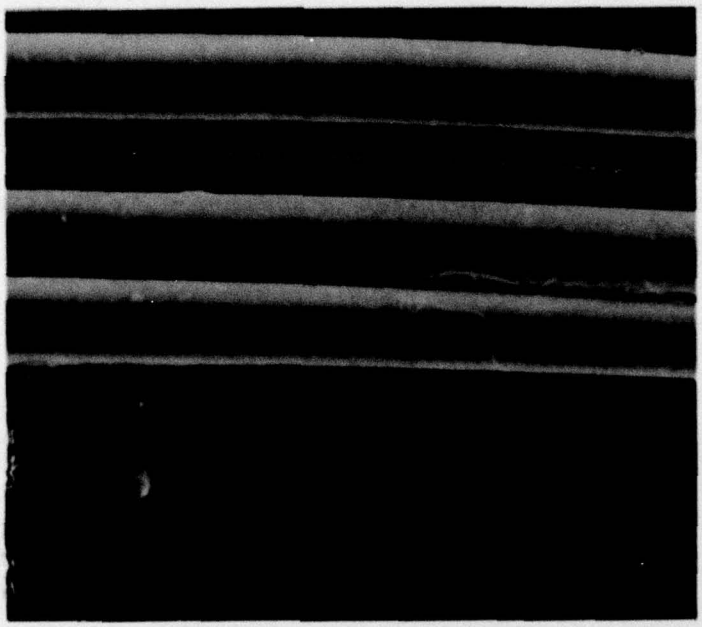
a. A high degree of nonuniformity and inhomogeneity resulting from the winding techniques employed (tooling, resin content, and resin cure)

b. Poor adhesion between fiber and matrix as a result of either the resin system, excessive fiber wetness during winding, or contaminated fibers

c. Excessive cracking of the matrix, especially in hoop wraps, resulting from cure stresses in the brittle epoxy, a poor cure schedule, or both

Several experiments were undertaken to address the effects of moisture and contamination on fiber-matrix bonding.

(a) SKIRT REGION (500 x) 20 μm



(b) POLAR REGION (1000 x) 10 μm

Fig. 11. Evidence of Fiber Wetting

III. EXPERIMENTAL FINDINGS

Findings of the failure analysis suggested the following experiments. The extreme brittleness of the resin system was felt to be responsible for the excessive cracking in the hoop windings, and the poor adhesion between fiber and matrix was thought to result from water either absorbed or adsorbed on the Kevlar fibers. The problem of excessive voids and poor layup was felt to result from poor winding procedures, which would probably improve with experience.

A. DENSITY MEASUREMENTS

Two approaches were used for checking the resin content of the SRM-1 prototype case, an actual fiber count of selected cross sections of the case and Archimedes' principle. Specimens measuring 0.5 in^2 were taken from the regions noted in Fig. 4. These specimens were weighed both in air and methanol, from which their densities were determined (Table 1). Assuming that no voids are present in the specimen, the weight percent resin is calculated by noting that

$$x = \frac{(\rho_k / \rho_c) - 1}{(\rho_k / \rho_r) - 1} \tag{1}$$

where x is the percent resin by weight, and ρ_k, ρ_r, and ρ_c are the densities of the Kevlar, resin, and composite respectively. Unfortunately, this technique is extremely sensitive to voids within the composite; the effect of a small percentage of voids may be approximated by

$$x = \frac{1 - (\rho_k / \rho_c)(1 - \epsilon)}{1 - (\rho_k / \rho_r)} \tag{2}$$

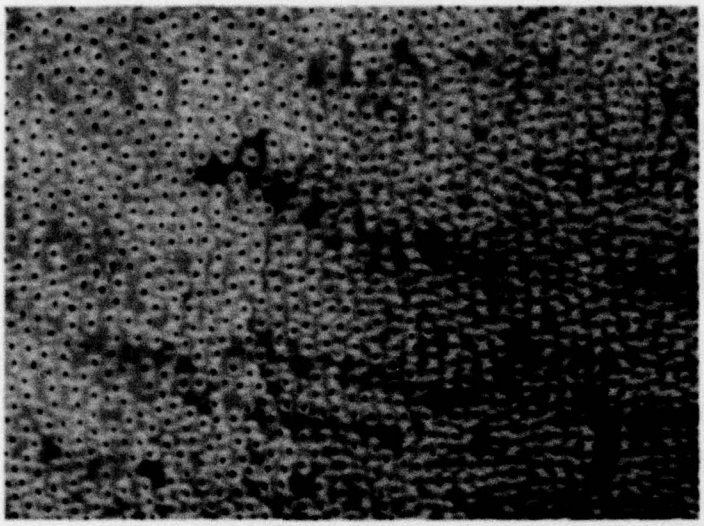
Table 1. Resin Content of SRM-1 Prototype

Region	Density, g/cm	Resin, wt%	Number of Fibers (Fig. 12)	Fiber, vol%	Resin, wt%
1	1.354	32	1073	63	33
2	1.347	35			
3	1.327	43			
6	1.327	43	1097	65	31
7	1.348	34			
Resin	1.200	100			
Kevlar	1.440	0			

where ϵ is the void fraction by volume. If it is assumed that regions 3 and 6, which gave the highest weight percent resin results, contain a 2 percent void, x is 32 percent instead of 43 percent; a 30 percent error in resin content thus results from a 2 percent volumetric void fraction. The sensitivity of this technique for determining resin content results from the similarity in density of the fiber and the resin and the fact that the densities of both are large relative to the density of the void.

The second approach for determining resin content consisted of carrying out an actual fiber count on polished cross sections taken from the skirt and dome regions of the SRM-1 prototype case (Fig. 12). To obtain a statistically meaningful approximation of the resin fraction, a relatively large (0.015 × 0.020 in.) cross section was measured in a randomly chosen area of the case. A total of 1097 of the 0.00047-in.-diameter fibers were counted in the skirt region and 1073 fibers were counted in the dome region; the dots evidenced near the center of each fiber indicates that it was included in the count. From these counts, the volume and weight fractions of the resin were calculated and are reported (Table 1).

A careful examination of Fig. 12b indicates that approximately 40 to 50 fibers could be placed in the void regions. This is equivalent to between



(a) DOME REGION: 1073 FIBERS (200 x)

(b) SKIRT REGION: 1097 FIBERS (200 x)

Fig. 12. Fiber Count on Polished Cross Sections

2 and 3 percent void space, which when substituted into Eq. (2) yields a good correlation between the two techniques. Due to the inhomogeneity of the composite, the actual fiber count technique yields the best results, and the areas used here should be considered minimal. Future studies should include counts from many areas of the case.

B. RESIN SYSTEM

The resin system used in the IUS 92-in. prototype motor case was reproduced for these experiments on filament winding resin cure and was prepared as follows:

Component	Parts by Weight
Epon 828 (epoxy resin)	65.0
ERL 4206 (cyclohexine diepoxide)	35.0
MDA (methylene dianiline)	39.5

and was cured by the following schedule:

16 hr at 140°F
1 hr at 200°F
4 hr at 280°F

where the rate of increase between each plateau is specified not to exceed 10°F/hour. This resin system (plus one variation) was used in all subsequent experiments.

C. WATER SORPTION

An inspection of the case winding facilities revealed that the Kevlar fibers had a residence time on the order of a minute between the dry box in which they were stored and the resin bath prior to winding. Since Kevlar fiber is known to be hygroscopic, thermal gravimetric analysis was used to

investigate the rate at which dry Kevlar fiber absorbs or adsorbs water. This instrument utilizes a quartz crystal microbalance to monitor the weight of a specimen, usually as a function of temperature. In this experiment the temperature was held constant, and the weight of Kevlar was monitored with time as the Kevlar was exposed first to vacuum and then to humid nitrogen (the actual percentage of water in the nitrogen atmosphere injected into the instrument is not known). The results shown (Fig. 13) indicate that dry Kevlar very rapidly picks up water. These results served to reinforce the suspicion that water might be responsible for the poor adhesion observed between fiber and resin.

D. FILAMENT WINDING

Water appeared to be chiefly responsible for the poor bonding between the resin and matrix; this hypothesis was tested by preparing composite specimens. Kevlar yarn was wound into Teflon spools and cured as described previously. The following specimens were made:

a. Kevlar yarn was wound onto a Teflon spool, vacuum dried at 200°F for three days, and vacuum impregnated in the resin bath for five minutes.

b. Kevlar yarn was wound onto a Teflon spool, exposed to +80 percent humidity for three days, and dip impregnated for one minute.

c. Kevlar yarn from a different (non-IUS) lot was used to prepare a vacuum impregnated specimen as described in (a) above.

After the standard cure cycles, the specimens were fractured and studied using the SEM (the Kevlar fiber as received was also studied). In every case the interface was found to be indistinguishable from those shown in Figs. 1b, 2, and 11b, which indicates that neither water nor contaminated Kevlar is responsible for the poor fiber-resin adhesion. The possibility that a resin component is responsible should be investigated; there is some indication that MDA leads to poor interaction with the polyaramide fiber.

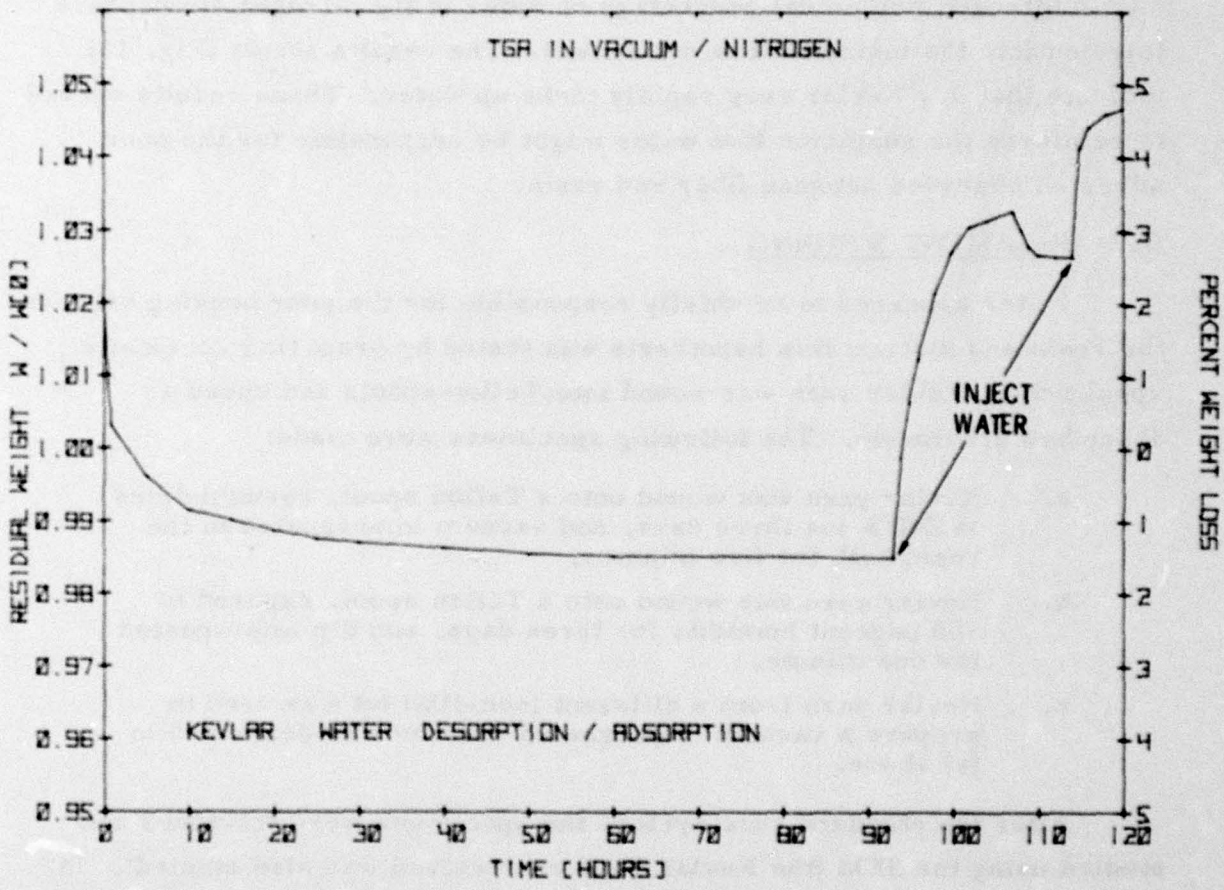


Fig. 13. Water Sorption Characteristics of Kevlar

E. RESIN CURE

The excessive cracking observed in the hoop wraps of the SRM-1 prototype may result from a poor cure schedule, the inherent brittleness of the MDA-cured Epon 828, or both. The rate of the curing reaction at 140°F was investigated using a differential scanning calorimeter (DSC). A sample of fresh resin was prepared and placed into a circulating air oven maintained at 140°F, and portions of the resin were subsequently removed at intervals of 0, 1, 2, 4, 8, and 144 hours and examined using the DSC.

The results of this study are shown in Fig. 14. If the reaction rate of the resin system at 140°F were sufficiently high, the exotherm in the vicinity of 170°F would rapidly diminish as the specimens were held at 140°F for longer periods of time. The total area under each exotherm is proportional to the total enthalpy of the reaction and is thus a measure of the extent (or percent completion) of the cure reaction. For example, when a second scan is made of specimen 1 (scan 1b), the exotherm does not appear, which indicates that the specimen was fully cured during the first scan. The result of measuring a very gradual decrease in the exotherm (Fig. 14) indicates that the cure reaction is not unstable at 140°F. Note that a quantitative comparison of these curves requires that they be normalized by the specimen mass; since specimen 3 is approximately one-third the mass of specimen 1, the total difference in area under the two exothermic peaks is not nearly as great as it appears at first glance.

The scans in Fig. 14b were separated from those of Fig. 14a because they were recorded at a different recorder attenuation (500 μV/in. in Fig. 14a vs 200 μV/in. in Fig. 14b). Further reduction of the data was not felt to be necessary for this purpose.

Specimens were also prepared, using the cure schedule reported previously, and examined using the DSC. The results indicated that the cure schedule was sufficient to fully cure these laboratory size specimens.

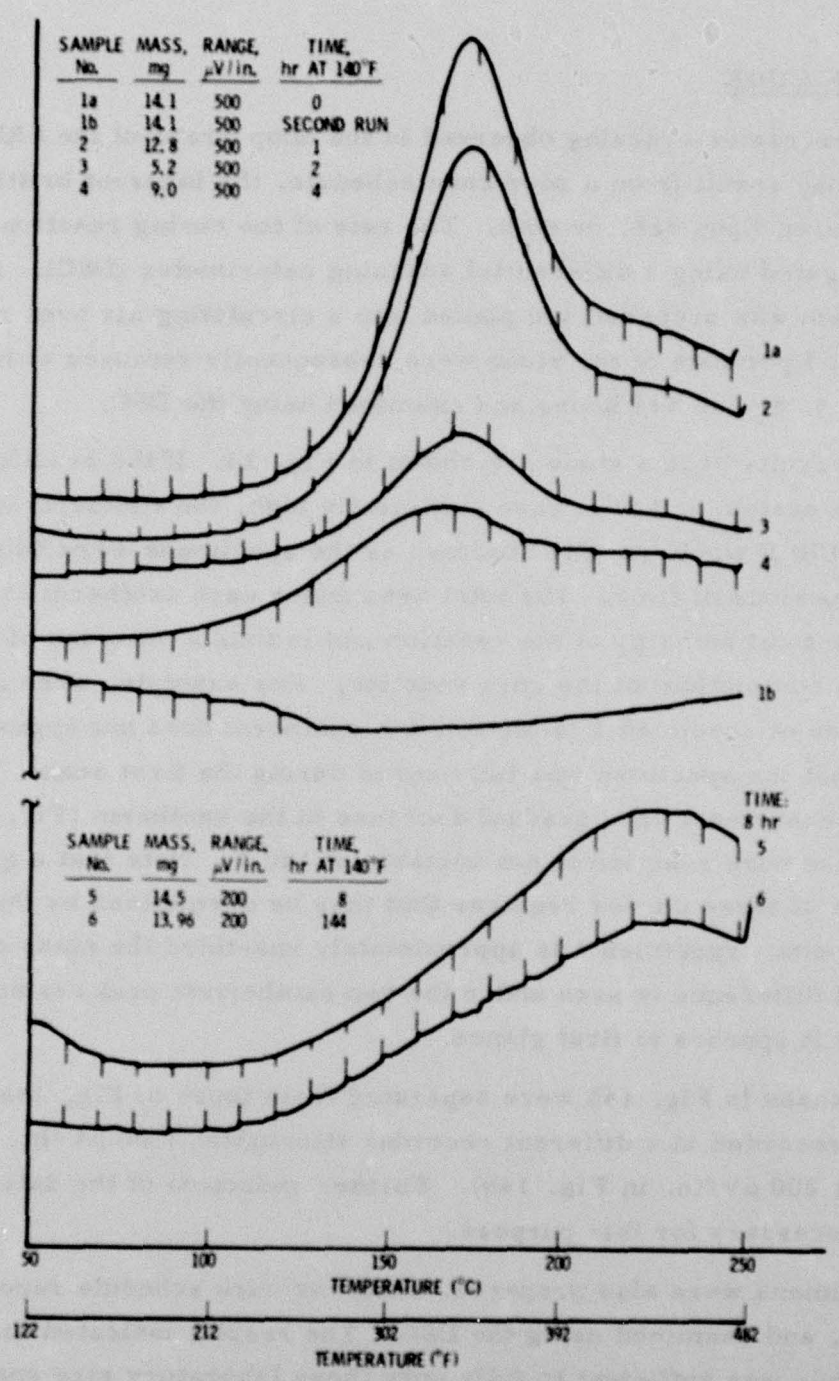


Fig. 14. DSC Studies of MDA-Cured Epon 828

The most alarming result of this study was the fact that the resin system gelled to a weak, brittle solid in three to four days at 70°F. This length of time would ordinarily pose no problem, but the SRM-1 prototype required seven days to wind, and this period will probably not be significantly shortened in the future. The interior of the motor case will thus become quite weak and brittle while the outer portion is still being wound. Additionally, resin flow that normally occurs during the initial phase of the cure cycle does not occur.

Although additional measurements indicate that the aged resin system attains its proper strength if it is later properly cured, it is feared that the gelled resin in the inner regions of the case may fracture due to its brittle, weak state as the stresses from the winding of the outer portion of the case are imparted to it. In any event, premature gelling leads to a nonuniform distribution of strain in the finished case and is probably partly responsible for the radial cracking evidenced in Fig. 7.

F. MECHANICAL TESTS OF RESIN

As reported in the preceding section, the resin system gells in three to four days at 70°F (a minimum of six days is currently required to wind the case). Additionally, the temperature during winding may reach 90°F, which further accelerates the gelling. Diametral compression, more commonly known as the Brazil test, was chosen as a simple technique for determining whether the delayed cure schedule affected the mechanical properties of the neat resin. Also included in this matrix was a resin system containing 20 percent MDA:

Cure Sample Component	Standard Cure 1[a]	Cure Delayed 7 Days 2[a]	Cure Delayed 7 Days 3[a]	Standard Cure 4[a]
Epon 828	65	65	65	65
ERL 4206	35	35	35	35
MDA	39.5	39.5	25	25

[a]Parts by weight

One-inch-diameter test tubes were used to cast epoxy rods from which 0.25-in.-thick Brazil test specimens were cut. As discussed previously, the cure cycle used did not include the 10°F/hr rate for temperature changes in the cure cycle; this oversight resulted in specimens that exhibited considerable internal strain when observed under cross polarizers. In fact, many of the epoxy rods were found to have fractured into halves when removed from the oven. All specimens exhibited a highly fractured surface condition, which was removed using a lathe prior to testing.

The critical step in the cure cycle is probably the heating from 140 to 200°F. Apparently, gelling has already occurred at 140°F, which leaves the resin quite hard, but the final cure (and final strength) does not occur until higher temperatures are reached (200 to 280°F). Rapid heating from 140 to 200°F results in thermal stress sufficient to fracture the one-inch-diameter rods.

The results of these tests are reported in Table 2. The specimens labeled PC received an additional post-cure of 16 hr at 280°F prior to testing to determine whether the standard cure was adequate. Within the scatter of the data, no significant differences in resin tensile strength are associated with a delay in cure schedule.

Table 2. Diametral Compression of Neat Resin

Group No.	σ_{ksi}	σ_{ksi} (PC)[a]
1	5.2	6.8
	6.1	7.3
	8.4	
2	5.3	6.7
	5.6	4.4
	5.4	4.9
3	6.7	
	5.9	
	6.6	
	6.4	
	6.5	
4	5.1	5.8
	4.8	5.4
	5.6	5.5

[a]The specimens received an additional cure: 16 hr at 280° F.

IV. CONCLUSIONS AND RECOMMENDATIONS

Optical microscopy demonstrated excessive voids and inhomogeneities throughout the SRM-1 prototype case. Much of this probably results from tooling and processing problems, which should improve as future cases are wound.

Lack of wetting, and consequently poor adhesion, between fiber and matrix was demonstrated by both optical and scanning electron microscopy, and it appears to be intrinsic to this particular resin system. Thermal gravimetric analysis indicated that dry Kevlar picks up water extremely rapidly. However, subsequent laboratory work showed that water was not the cause of the poor wetting of this resin system. Additional investigation of epoxy resins that employ either aramide or an amine curing system as a means of improving adhesion should be carried out. There is evidence that long, cylindrical Kevlar filament-wound cases do not rely on good adhesion between fiber and matrix; however, these cases are designed with full knowledge of this effect. In the short IUS case, the lack of good adhesion may lead to undesired effects.

Epon 828/ERL 4206/MDA epoxy resin is a relatively rigid, low-strain-to-failure matrix. Consequently, optimum utilization of a high modulus, low-strain-to-failure Kevlar fiber demands that maximum uniformity in strain in the composite system be achieved during manufacturing. There simply is no mechanism for relieving local strain, other than irreversible fracture, available to the system. Consequently, manufacturing that requires winding times longer than the gelling period of the resin system and manufacturing that allows excessive temperature excursions (leading to strain inhomogeneities through the composite) cannot produce an optimum motor case. Attention should therefore be directed toward winding the case in a shorter period of time or prolonging the potlife of the resin.

Additionally, temperature must be controlled during winding to minimize the inhomogeneities in strain that result from thermal expansion of the tooling and Kevlar fiber during winding.

LABORATORY OPERATIONS

The Laboratory Operations of The Aerospace Corporation is conducting experimental and theoretical investigations necessary for the evaluation and application of scientific advances to new military concepts and systems. Versatility and flexibility have been developed to a high degree by the laboratory personnel in dealing with the many problems encountered in the nation's rapidly developing space and missile systems. Expertise in the latest scientific developments is vital to the accomplishment of tasks related to these problems. The laboratories that contribute to this research are:

Aerophysics Laboratory: Launch and reentry aerodynamics, heat transfer, reentry physics, chemical kinetics, structural mechanics, flight dynamics, atmospheric pollution, and high-power gas lasers.

Chemistry and Physics Laboratory: Atmospheric reactions and atmospheric optics, chemical reactions in polluted atmospheres, chemical reactions of excited species in rocket plumes, chemical thermodynamics, plasma and laser-induced reactions, laser chemistry, propulsion chemistry, space vacuum and radiation effects on materials, lubrication and surface phenomena, photosensitive materials and sensors, high precision laser ranging, and the application of physics and chemistry to problems of law enforcement and biomedicine.

Electronics Research Laboratory: Electromagnetic theory, devices, and propagation phenomena, including plasma electromagnetics; quantum electronics, lasers, and electro-optics; communication sciences, applied electronics, semiconducting, superconducting, and crystal device physics, optical and acoustical imaging; atmospheric pollution; millimeter wave and far-infrared technology.

Materials Sciences Laboratory: Development of new materials; metal matrix composites and new forms of carbon; test and evaluation of graphite and ceramics in reentry; spacecraft materials and electronic components in nuclear weapons environment; application of fracture mechanics to stress corrosion and fatigue-induced fractures in structural metals.

Space Sciences Laboratory: Atmospheric and ionospheric physics, radiation from the atmosphere, density and composition of the atmosphere, aurorae and airglow; magnetospheric physics, cosmic rays, generation and propagation of plasma waves in the magnetosphere; solar physics, studies of solar magnetic fields; space astronomy, x-ray astronomy; the effects of nuclear explosions, magnetic storms, and solar activity on the earth's atmosphere, ionosphere, and magnetosphere; the effects of optical, electromagnetic, and particulate radiations in space on space systems.

THE AEROSPACE CORPORATION
El Segundo, California

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